Materials and Design 36 (2012) 694–698

Contents lists available at SciVerse ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Effects of heat treatment parameters on the microstructure and mechanical properties of *in situ* TiBw/Ti6Al4V composite with a network architecture

L.J. Huang *, H.Y. Xu, B. Wang, Y.Z. Zhang, L. Geng

National Key Laboratory of Science and Technology on Precision Heat Processing of Metals, Harbin Institute of Technology, P.O. Box 433, Harbin 150001, China

ARTICLE INFO

Article history:
Received 1 November 2011
Accepted 9 December 2011
Available online 17 December 2011

Keywords:
A. Composites
C. Heat treatments
E. Mechanical properties

ABSTRACT

In situ 5 vol.% TiB whiskers reinforced Ti6Al4V (TiBw/Ti64) composites with a network architecture were heat treated with different parameters in order to further improve their mechanical properties. The microstructure results showed that the fraction of the transformed β phase in the matrix increases with increasing quenching temperatures, which leads to a significant improvement in strength at room and high temperatures. Both the size and fraction of fine α + β phases transformed from martensite phase increase with increasing aging temperatures, which decreases the strength of the composite. When the testing temperature is lower than 600 °C, the strength of the 5 vol.%TiBw/Ti64 composite is significantly enhanced by heat treatment consisting of water quenching and aging processes with proper parameters. This can be attributed to the superior heat treatment strengthening effect on the basis of the network boundary strengthening effect.

1. Introduction

As a typical member of metal matrix composites (MMCs) family, titanium matrix composites (TMCs) offer a combination of good mechanical properties and high temperature durability that render them attractive materials for automotive, aerospace and military applications [1–5]. Discontinuously reinforced titanium matrix composites (DRTMCs) with a homogenous microstructure, especially those fabricated by *in situ* methods such as powder metallurgy (PM) and cast techniques are sought-after due to their superior properties and low cost [6–10]. However, experimental results in the past 40 years have adequately demonstrated that the composites with a homogeneous reinforcement distribution just can exhibit a limited improvement of mechanical properties, even inferior mechanical properties such as extreme brittleness for the TMCs fabricated by conventional powder metallurgy (PM) [11–15]. Maybe, that is why the previous researches have always focused on the microstructure, hardness, high temperature performance and compressive property without tensile property [6,12]. Tjong et al. [12] has concluded that *in situ* processing techniques offer several advantages such as easy fabrication, lower energy consumption and higher purity of ceramic reinforcements produced comparing to the *ex situ* route. Fortunately, in our previous work [1,2,16–18], not only the strength but also the ductility of the *in situ* DRTMCs is significantly improved by tailoring a novel network reinforcement architecture compared with those of DRTMCs with a homogeneous reinforcement distribution. This work echoes a recent proposal by Lu [19] that the overall properties of composites can be further enhanced by assembling metals with other components in a controlled way to form novel multi-scale hierarchical structures, compared with a conventional or homogeneous composite structure.

Furthermore, the subsequent treatment can further improve the mechanical properties of MMCs [20–24]. Li et al. [22] pointed out that the tensile strength of (TiB + La_2O_3)/Ti composites increases while the ductility decreases sharply by heat treatment. The heat treatment including solid solution and aging processes, as one of the effective strengthening treatments, can further improve the strength of DRTMCs by strengthening the titanium alloy matrix [22–25]. Furthermore, Mceldowney et al. [23] also pointed out that the highest strength can be obtained by solution and aging processes. Strength is one of the most important characteristics for structural applications for DRTMCs [4]. However, there is little study on the heat treatment strengthening DRTMCs in the past 40 years. The main reason is that the conventional titanium alloy matrix composites with a homogenous microstructure, especially DRTMCs fabricated by PM always exhibit a very low ductility, even extreme brittleness [12–14]. In our previous work, not only the strength but also the ductility of 5 vol.%TiBw/Ti64 composites is significantly improved by tailoring a novel network microstructure [1,2]. Therefore, it is important and necessary to investigate the effects of subsequent heat treatment on these novel composites in order to further improve their mechanical properties.

* Corresponding author. Tel.: +86 451 86418836; fax: +86 451 86413922.
E-mail addresses: ljhuanghit@yahoo.com.cn, huanglujun@hit.edu.cn (L.J. Huang).

0261-3069/$ - see front matter Crown Copyright © 2011 Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.matdes.2011.12.021

2. Experimental procedures

2.1. Composite fabrication

As reported in our previous work [1,2], 5 vol.%TiBw/Ti64 composite with a novel network reinforcement architecture was successfully fabricated by selecting the raw materials consisting of the large Ti64 powders and fine TiB_2 powders and processing by low-energy milling and reaction hot pressing. Fig. 1 shows the network microstructure of the typical 5 vol.%TiBw/Ti64 (200 μm) composite [2]. The synthesized TiB whisker reinforcements were distributed around Ti64 matrix particles and formed a three dimensional (3D) equiaxed network microstructure. Ti64 matrix consists the grey α phase and the bright β phase which is located between α phases.

In order to further investigate the effects of the subsequent heat treatment on the microstructure and mechanical properties of TiBw/Ti64 composites with a novel network microstructure, heat treatment was performed on the typical 5 vol.%TiBw/Ti64 (200 μm) composite with a network microstructure. Water quenching (WQ) was carried out at 840–930 °C and aging at 500–580 °C, which corresponds to the heat treatment parameters of Ti64 alloy shown in Fig. 2 [26]. As shown in Fig. 2, the α phase of Ti64 alloy begins to transform to β phase at 600 °C and thoroughly transforms to β phase at 985 °C. When the quenching temperature is higher than 800 °C, partial β phase can transform to martensite phase. Therefore, the quenching temperature has been selected higher than 800 °C, and the aging temperature lower than 600 °C.

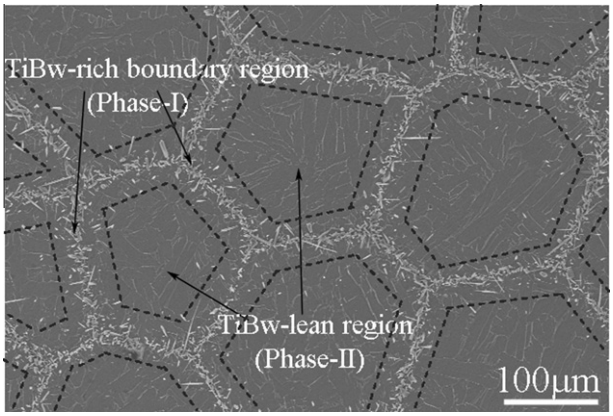


Fig. 1. SEM micrograph of the *in situ* 5 vol.%TiBw/Ti64 composite with a network TiBw distribution [2].

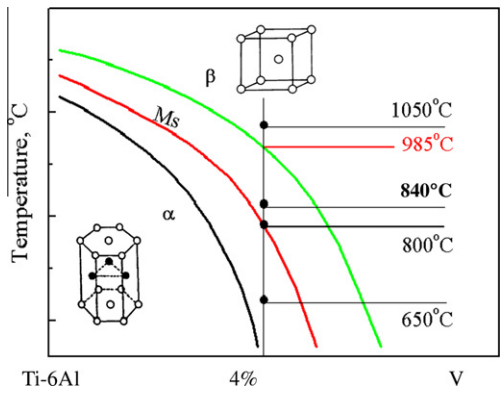


Fig. 2. Schematic illustration of martensitic phase transformation of Ti64 alloy [26].

Microstructural characterizations were performed using a scanning electron microscope (SEM, Hitachi S-4700). Room temperature and high temperature tensile tests, which refers to the metal materials testing standards of ISO 6892: 1998 [27] and ISO 783: 1999 [28], were carried out using an Instron-1186 universal testing machine at a constant crosshead speed of 0.5 mm/min (approximate strain rate is 5.0×10^{-4}/s). A total of five tensile samples with dimensions of 15 mm × 5 mm × 2 mm were tested for each sample.

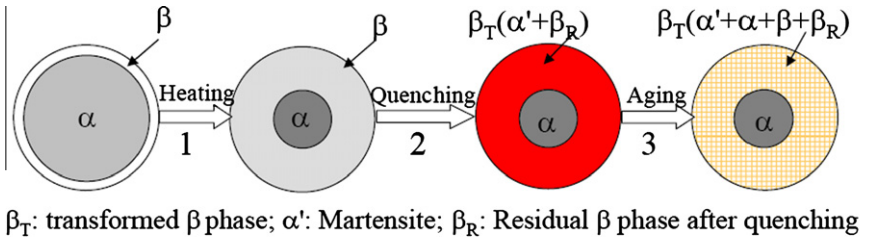
3. Results and discussions

3.1. Microstructure

Fig. 3 shows the schematic illustrations of the microstructure evolution of Ti64 alloy during different heat treatment processes. It can be seen that the primary α phase is converted into hot temperature β phase during heating process (Figs. 2 and 3), and the fraction of β phase increases with increasing the heating temperatures. During this process, the content of Al element in both the decreased α phase and the increased β phase increases due to the decreasing fraction of α phase [26]. In the following WQ process, martensite α' phase is formed from the high temperature β phase, and the fraction of martensite phase increases with increasing the quenching temperatures [22–26]. It is certain that there is a partial residual β phase remained [26]. The martensite phase and the residual β phase compose the transformed β phase, marked as β_T. During the final aging process, a partial martensite α' phase disintegrates into fine α + β phases, and the fraction and the size of the fine α + β phases increase with increasing the aging temperature [22–24].

Fig. 4 shows the SEM micrographs of the composite samples by WQ at 840 °C and 870 °C followed by aging at 500 °C for 6 h. After heat treatment, the bright β phase in the previous Ti64 matrix is converted into the transformed β phase, which consists of the residual β phase, martensite α' phase formed in the WQ process and the disintegrated fine α + β phases transformed during the aging process (Figs. 3 and 4). Comparing Fig. 4 with Fig. 1, the volume fraction of β_T in the as-heat treated composite is much higher than that of the stable β phase in the original composite. Fig. 4 also indicates that the volume fraction of β_T increases with increasing the WQ temperature. These phenomena are consistent with the schematic illustrations of Ti64 alloy (Fig. 3). In Mceldowney's work [23], the authors also found that the α grain size, volume fraction and morphology of primary α phase, transformed β phase can be influenced by heat treatment with different parameters. After the heat treatments, the increasing transformed β phase must lead to the enhancement of the hardness and strength of the Ti64 matrix in the present work according to the previous similar system [22,23]. In addition, the contrast between the primary α phase and the transformed β phase decreases with increasing the quenching temperature, which indicates the increasing fraction of the martensite and the decreasing fraction of the residual β phase in β_T. Increasing the fraction of the martensite is beneficial to the hardness and the strength of the composite [22–25].

Fig. 5 shows the SEM micrographs of 5 vol.%TiBw/Ti64 composite undergone WQ at 900 °C and aging at 500 °C and 580 °C for 6 h. As shown in Fig. 5, the volume fraction of β_T has no evident change due to the same quenching temperature. However, the contrast between the primary α phase and the transformed β phase increases with increasing the aging temperature, which can be attributed to that the disintegrated fine α + β phase transformed from martensite increases with increasing the aging temperatures as shown in Figs. 3 and 5. Moreover, the size of the fine α + β phase increases with increasing the aging temperature, which is consistent with



β_T: transformed β phase; α': Martensite; β_R: Residual β phase after quenching

1: Al content in βand α phases increase with increasing the heating temperature.
2: The fraction of α' phase increases with increasing the heating/quenching temperature.
3: The fraction and the size of the fine α+β phases increase with increasing the aging temperature.

Fig. 3. The schematic illustrations of microstructure evolution of Ti64 alloy during the heat treatments.

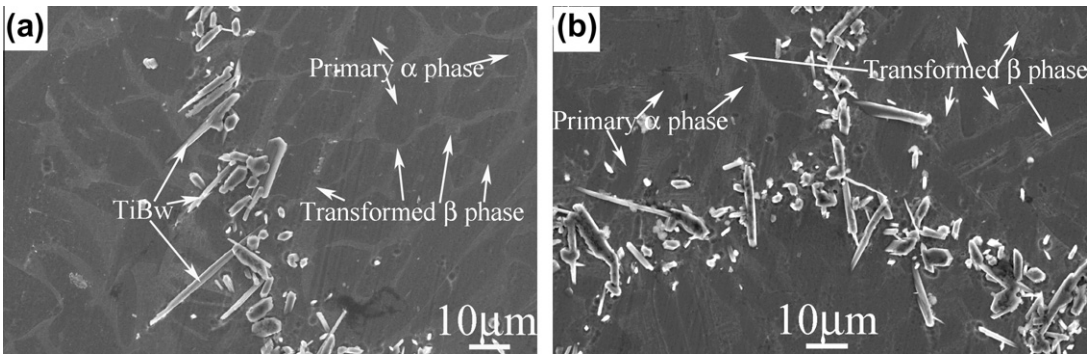


Fig. 4. SEM micrographs of 5 vol.%TiBw/Ti64 composite undergone WQ at (a) 840 °C and (b) 870 °C and aging at 500 °C for 6 h.

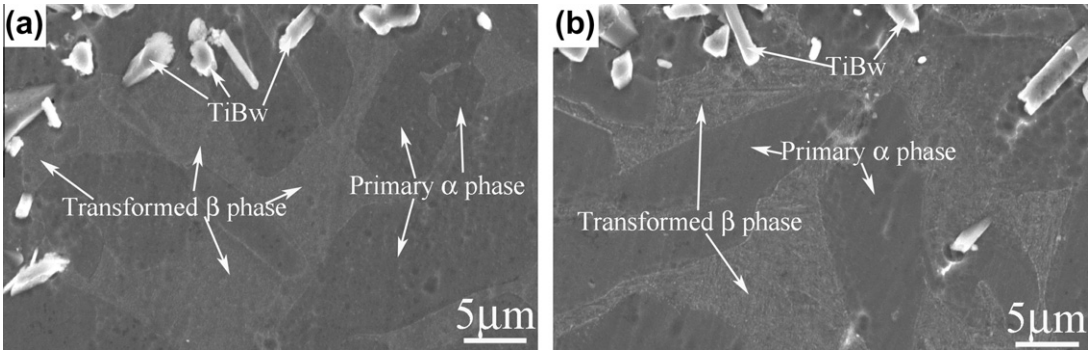


Fig. 5. SEM micrographs of 5 vol.%TiBw/Ti64 composite undergone WQ at 900 °C and aging at (a) 500 °C and (b) 580 °C for 6 h.

Li's work [23]. Increasing the disintegrated α + β phase is beneficial to the combination properties but harmful to the hardness of the composites.

In addition, there is no change for the TiBw reinforcement during the heat treatment process as shown in Figs. 4 and 5. According to Li's work [22], that is due to the relative low temperature for ceramic reinforcement: no any other phase is formed and no interfacial reaction is observed during heat treatment process. This phenomenon also indicates that TiB whisker reinforcement and the composites are stable. Therefore, the effects of heat treatment on the composites are mainly performed to the Ti64 matrix [20–24], which is similar with that of the monolithic Ti64 alloy [25].

3.2. Mechanical properties

Fig. 6 shows the variations of room temperature tensile strength and elongation of the 5 vol.%TiBw/Ti64 composite with increasing the quenching temperatures. Firstly, the tensile strength primarily

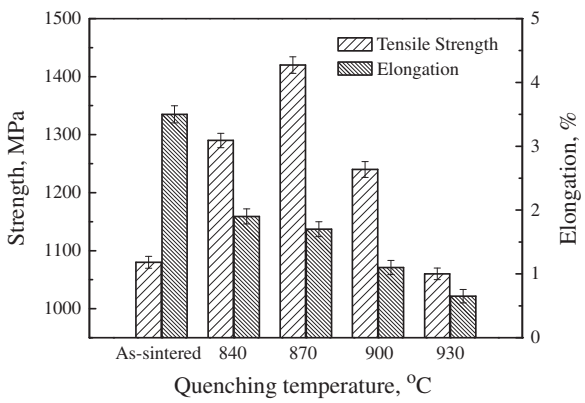


Fig. 6. Variations of tensile strength and elongation of 5 vol.%TiBw/Ti64 composite with increasing the quenching temperatures followed by aging at 500 °C.

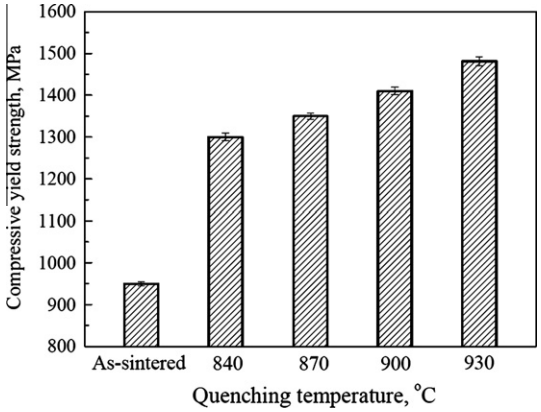


Fig. 7. Variation of compressive yield strength of 5 vol.%TiBw/Ti64 composite with increasing the quenching temperatures.

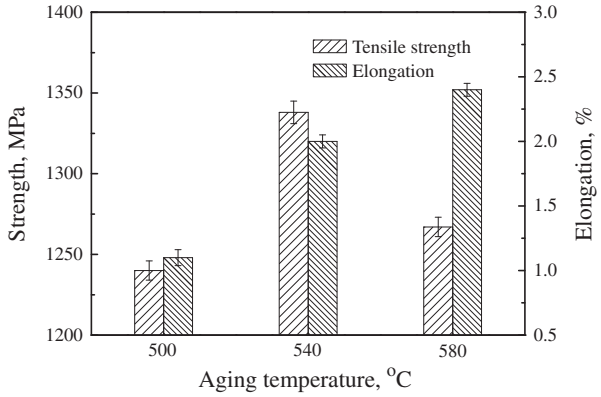


Fig. 8. Variation of the room temperature tensile properties of 5 vol.% TiBw/Ti64 composite with increasing the aging temperatures after WQ at 900 °C.

increases and then decreases, however, the elongation always decreases with increasing the quenching temperature. Secondly, compared with that of the as-sintered composite [1], the tensile strength of the composite quenched at 870 °C is increased from 1090 MPa to 1423 MPa. In other words, the tensile strength is increased by 30.6% by WQ at 870 °C and aging at 500 °C, which can be reviewed as a significant improvement for DRTMCs in strength at room temperature [12,22,25]. This is consistent with Mceldowney's work [23]: the highest strength can be obtained by solution and aging processes. As mentioned above, the increased strength and the decreased elongation can be attributed to the increased volume fraction of martensite phase/transformed β phase [23,24] and the increased content of Al element in both the transformed β phase and the remained α phase (Fig. 3) [26]. Then, the decreasing strength may be due to the excessive martensite or hardness, which is generated by much high quenching temperatures [22].

Fig. 7 shows the variation in the compressive yield strength of the composites in order to further demonstrate the strength variation. It is obvious that the compressive yield strength constantly increases with increasing the quenching temperature. Even, the yield strength is close to 1500 MPa after solution at 930 °C followed by aging at 500 °C. It is worth pointing out that the ultimate strength should be much higher than the yield strength, which can be viewed as the most effective strengthening process up to date for DRTMCs [1,2,12]. This phenomenon also indicates that the decrease of tensile strength shown in Fig. 6 is due to the excessive hardness and low ductility of the matrix, which is consistent with Figs. 3 and 6. Therefore, the strength increases but the ductility decreases with increasing the quenching temperatures.

In order to further detect the effect of the aging temperature, the samples were quenched at 900 °C followed by aging at 500 °C, 540 °C and 580 °C, respectively. As shown in Fig. 8, the elongation increases with increasing the aging temperatures, which is due to the increasing volume fraction of the fine α + β phases disintegrated from the quenching martensite. That is why aging treatment can effectively improve the ductility of the as quenched TMCs [21–23]. The increasing elongation should correspond to the decreasing strength. However, the strength of the sample aged at 540 °C is higher than that of the sample aged at 500 °C, which is due to the excessive hardness for the composite quenched at 900 °C. This phenomenon also indirectly demonstrates that the decreasing strength shown in Fig. 6 is caused by the quenching treatment at the overtop temperatures. This phenomenon also indicates that the tensile properties of the composites can be controlled by not only the quenching temperatures but also the aging temperatures [21–24].

Fig. 9 shows the high temperature tensile stress–strain curves of the 5 vol.%TiBw/Ti64 composite treated by 930 °C/40 min/WQ + 500 °C/6 h/AC. As shown in Fig. 9, the tensile strength of the composite is significantly increased close to 1000 MPa and 900 MPa at 400 °C and 500 °C, respectively. However, the strength sharply decreases to 580 MPa and 330 MPa when the test temperature is up to 600 °C, and then 700 °C. This is mainly due to the matrix softening [26]. Comparing with that of the as-sintered composites, the tensile strength of the as-heat treated composites is significantly increased below 500 °C, while the tensile strength is just slightly increased above 600 °C. The main reasons for the above phenomena can be interpreted as following: the formation of martensite phase play the effective strengthening effect at 400 °C and 500 °C. However, the martensite phase is quickly transformed into α + β phases (Fig. 3), even the α + β phases are quickly coarsened at 600 °C and 700 °C. In other words, the strengthening effect introduced by the martensite phase remarkably weakens, and even disappears over 600 °C, however, the strengthening effect introduced by the increase of Al element content in α phase can also play a limited strengthening effect.

For the significant improvement in strength of DRTMCs, on the one hand, the improvement in the strength by heat treatment can be mainly attributed to the following four factors: the formation of martensite, the increased transformed β phase, the increased Al element content in the remained α phase and the formation of the fine α + β phases as mentioned above [21–23]. On the other hand, the excellent strength (1423 MPa and 1500 MPa) of 5 vol.%-TiBw/Ti64 composite with a network microstructure depends on

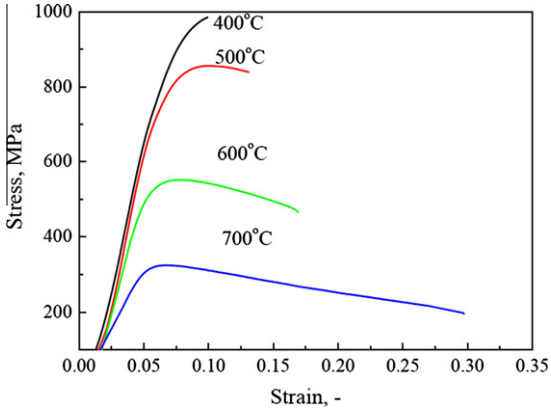


Fig. 9. The stress–strain curves of 5 vol.%TiBw/Ti64 composite heat treated at 930 °C/WQ + 500 °C/6 h/AC.

both the superior network boundary strengthening effect and the heat treatment strengthening effect, simultaneously [1,2].

4. Conclusions

The effects of heat treatment on the microstructure and mechanical properties of the 5 vol.%TiBw/Ti64 composite with a network microstructure have been investigated. The present work has led to the following findings:

(1) The volume fraction of the transformed β phase consisting of the residual β phase and martensite α' phase increases with increasing the WQ temperatures, and the disintegrated fine α + β phases increase with increasing the aging temperatures.
(2) The strength and hardness increase with increasing the WQ temperatures due to the increasing martensite phase, however, they decrease with increasing the aging temperatures due to the increasing disintegrated fine α + β phases.
(3) The room temperature tensile strength of 5 vol.%TiBw/Ti64 composite can be increased from 1090 MPa to 1432 MPa by the heat treatment of 870 °C/40 min/WQ + 500 °C/6 h/AC. The tensile strength at 400 °C, 500 °C and 600 °C can be increased to 985 MPa, 848 MPa and 567 MPa, respectively, by the heat treatment of 930 °C/40 min/WQ + 500 °C/6 h/AC due to the superior heat treatment strengthening effect on the basis of the network boundary strengthening effect.

Acknowledgements

This work is financially supported by the National Natural Science Foundation of China (NSFC) under Grant No. 51101042, the Fundamental Research Funds for the Central Universities under grant no. HIT. NSRIF. 201131, the 50th-class General Financial Grant from the China Postdoctoral Science Foundation under grant No. 2011M500653 and the Royal Society (RS)-NSFC International Joint Project under Grant No. 51011130206.

References

[1] Huang LJ, Geng L, Li AB, Yang FY, Peng HX. In situ TiBw/Ti–6Al–4V composites with novel reinforcement architecture fabricated by reaction hot pressing. Scr Mater 2009;60(11):996–9.
[2] Huang LJ, Geng L, Peng HX, Zhang J. Room temperature tensile fracture characteristics of in situ TiBw/Ti6Al4V composites with a quasi-continuous network architecture. Scr Mater 2011;64(9):844–7.
[3] Tjong SC, Ma ZY. Microstructural and mechanical characteristics of in situ metal matrix composites. Mater Sci Eng R 2000;29(3–4):49–113.
[4] Miracle DB. Metal matrix composites – from science to technological significance. Comp Sci Tech 2005;65(15–16):2526–40.
[5] Huang B, Yang Y, Luo H, Yuan M. Effects of the coating system and interfacial region thickness on the thermal residual stresses in SiCf/Ti–6Al–4V composites. Mater Des 2009;30:718–22.
[6] Liu D, Zhang SQ, Li A, Wang HM. Creep rupture behaviors of a laser melting deposited TiC/TA15 in situ titanium matrix composite. Mater Des 2010;31:3127–33.
[7] Ma FC, Lu WJ, Qin JN, Zhang D, Ji B. The effect of forging temperature on microstructure and mechanical properties of in situ TiC/Ti composites. Mater Des 2007;28:1339–42.
[8] Wang MM, Lu WJ, Qin JN, Ma FC, Lu JQ, Zhang D. Effect of volume fraction of reinforcement on room temperature tensile property of in situ (TiB + TiC)/Ti matrix composites. Mater Des 2006;27:494–8.
[9] Boehlert CJ, Tamirisakandala S, Curtin WA, Miracle DB. Assessment of in situ TiB whisker tensile strength and optimization of TiB-reinforced titanium alloy design. Scr Mater 2009;61:245–8.
[10] Panda KB, Ravi Chandran KS. First principles determination of elastic constants and chemical bonding of titanium boride (TiB) on the basis of density functional theory. Acta Mater 2006;54:1641–57.
[11] Ni DR, Geng L, Zhang J, Zheng ZZ. Fabrication and tensile properties of in situ TiBw and TiCp hybrid-reinforced titanium matrix composites based on Ti–B4C–C. Mater Sci Eng A 2008;478:291–6.
[12] Tjong SC, Mai YW. Processing-structure-property aspects of particulate-and whisker-reinforced titanium matrix composites. Comp Sci Tech 2008;68(3–4):583–601.
[13] Morsi K, Patel VV. Processing and properties of titanium–titanium boride (TiBw) matrix composites-a review. J Mater Sci 2007;42(6):2037–47.
[14] Feng HB, Zhou Y, Jia DC, Meng QC. Microstructure and mechanical properties of in situ TiB reinforced titanium matrix composites based on Ti–FeMo–B prepared by spark plasma sintering. Comp Sci Tech 2004;64(16):2495–500.
[15] Sen I, Tamirisakandala S, Miracle DB, Ramamurty U. Microstructural effects on the mechanical behavior of B-modified Ti–6Al–4V alloys. Acta Mater 2007;55(15):4983–93.
[16] Huang LJ, Geng L, Peng HX, Balasubramaniam K, Wang GS. Effects of sintering parameters on the microstructure and tensile properties of *in situ* TiBw/Ti6Al4V composites with a novel network architecture. Mater Des 2011;32(6):3347–53.
[17] Huang LJ, Geng L, Peng HX. In-situ (TiBw + TiCp)/Ti6Al4V composites with a network reinforcement distribution. Mater Sci Eng A 2010;527(24–25):6723–7.
[18] Huang LJ, Geng L, Xu HY, Peng HX. In situ TiC particles reinforced Ti6Al4V matrix composite with a network reinforcement architecture. Mater Sci Eng A 2011;528(6):2859–62.
[19] Lu K. The future of metals. Science 2010;328:319–20.
[20] Hill D, Banerjee RB, Huber D, Tiley J, Fraser HL. Formation of equiaxed alpha in TiB reinforced Ti alloy composites. Scripta Mater 2005;52(5):387–92.
[21] Rastegari HA, Asgari S, Abbasi SM. Producing Ti–6Al–4V/TiC composite with good ductility by vacuum induction melting furnace and hot rolling process. Mater Des 2011;32:5010–4.
[22] Li JX, Wang LQ, Lu WJ, Zhang D, et al. The effect of heat treatment on thermal stability of Ti matrix composite. J Alloy Comp 2009;509(1):52–6.
[23] Mceldowney DJ, Tamirisakandala S, Miracle DB. Heat-treatment effects on the microstructure and tensile properties of powder metallurgy Ti–6Al–4V alloys modified with boron. Metall Mater Trans A 2010;41:1003–15.
[24] Zhang ZG, Qin JN, Lu WJ, Zhang D, et al. Effect of β heat treatment temperature on microstructure and mechanical properties of in situ titanium matrix composites. Mater Des 2010;31:4269–73.
[25] Gorsse S, Miracle DB. Mechanical properties of Ti–6Al–4V/TiB composites with randomly oriented and aligned TiB reinforcements. Acta Mater 2003;51:2427–42.
[26] Welsch G, Boyer R, Collings EW. Materials properties handbook: titanium alloys. ASM International; 1994.
[27] ISO 6892. Metallic materials-tensile testing at ambient temperature; 1998.
[28] ISO 783. Metallic materials-tensile testing at elevated temperature; 1999.